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(54) Title: PACKAGED AGROCHEMICAL COMPOSITION

(57) Abstract

An agrochemical composition packaged in a water-soluble or water-dispersible sachet, the agrochemical composition comprising a water-soluble, agrochemically active ingredient, water and an agent to minimise water loss through the walls of the sachet.

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PACKAGED AGROCHEMICAL COMPOSITION

The present invention relates to a packaged agrochemical composition comprising a water-soluble, agrochemically active ingredient, the composition being packaged in a water-soluble or water-dispersible sachet.

A gel (comprising a hazardous product, a surfactant, an acrylic acid polymer or copolymer and water) which is suitable for packaging in a water-soluble sachet is disclosed in WO92/01377. An aqueous composition, comprising a hazardous product, an electrolyte and water and packaged in a water-soluble bag, is disclosed in EP-A1-0518689. It has been found that, when the aqueous compositions of the prior art are packaged in water-soluble sachets (such as sachets made from polyvinyl alcohol), water from the composition permeates through the wall of the sachet. The water that permeates can affect the outer surface of the sachet (for example by causing the bag to dissolve or adhere to the secondary packaging) and, thereby, reduce the shelf-life of the packaged product.

Water-soluble films comprising a barrier coating are disclosed in US3186869, US3322674 and GB-954602, whilst water-soluble films comprising a layer of particulate inert plastics material having high water repellency are disclosed in EP-A2-0079248.

The present invention provides an agrochemical composition packaged in a water-soluble or water-dispersible sachet, the agrochemical composition comprising a water-soluble, agrochemically active ingredient, water and an agent to minimise water loss through the walls of the sachet.

In one aspect the present invention provides an agrochemical composition packaged in a water-soluble sachet, the agrochemical composition comprising a water-soluble, agrochemically active ingredient, water and an agent to minimise water loss through the walls of the sachet.

In a further aspect the present invention provides an agrochemical composition packaged in a water-soluble or water-dispersible sachet obtained by packing a composition comprising a water-soluble, agrochemically active ingredient, water and an agent to minimise water loss through the walls of the sachet in a water-soluble or water-dispersible sachet.

The agent to minimise water loss through the walls of the sachet is, for example, an alkyl, alkenyl, aryl or arylalkyl acid or a salt or ester thereof; an ester of a naturally occurring oil; a mineral or synthetic oil; an alcohol, an ether of an alcohol or a glyceride. It is preferred

that the agent is an ester of an alkyl, alkenyl, aryl or arylalkyl acid; an ester of a naturally occurring oil; or a mineral or synthetic oil.

Aryl, and the aryl moiety of arylalkyl, is, preferably, phenyl. Aryl (especially phenyl) is optionally substituted with alkyl, alkenyl, alkynyl, phenylalkyl, phenylalkenyl or phenylalkynyl.

Alkyl groups are straight or branched chain and preferably contain from 1 to 24 carbon atoms. Alkyl is, for example, octyl, nonyl, decyl or dodecyl.

The alkyl group of arylalkyl or phenylalkyl is straight or branched chain and preferably contains from 1 to 20 (for example 1 to 10) carbon atoms.

Alkenyl groups, and the alkenyl moiety of phenylalkenyl, are straight or branched chains and preferably contain from 2 to 24, especially from 10 to 20, carbon atoms and 1, 2 or 3 double bonds. Alkenyl is, for example, stearyl, linolenyl, linolyl, licosenyl, erucyl, palmitolyl, oleyl or undecenyl.

Alkynyl groups, and the alkynyl moiety of phenylalkynyl, are straight or branched chains and preferably contain from 2 to 24, especially from 10 to 20, carbon atoms and 1, 2 or 3 triple bonds.

Suitable acid groups include carboxylic acids and sulphonic acids.

Salts include alkali metal salts such as sodium or potassium salts.

Esters include alkyl esters.

Naturally occurring oils include oils extracted from plants, seeds, nuts animals or fish and these are, for example, canola, sunflower or peanut oil. Such oils are, essentially, mixtures of esters of several unsaturated carboxylic acids. For example, sunflowerate includes esters of C18 unsaturated carboxylic acids. Other naturally occurring oils include derivatives of stearic, linolenic, linolic, licosenic, erucic, palmitic, oleic or undecenic acid.

Mineral oils include paraffin oils. Suitable mineral oils include, for example, ISOPAR® M or EXXSOL®-D110 (both available from EXXON), or SUNSPRAY® 6N or SUNSPRAY® 11N (both available from Sun Lubricants Limited, UK).

Synthetic oils include oils comprising C_{6-24} alkyl, C_{6-24} alkenyl or a fatty acid derivative (such as a derivative (especially an ester, such as a C_{1-6} alkyl ester) of stearic, linolenic, linolic, licosenic, erucic, palmitic, oleic or undecenic acid).

Alcohols include straight or branched chain alcohols (such as glycerols) while ethers of alcohols include ethers of oleyl alcohol.

The water-soluble agrochemically active ingredient, is, for example, a herbicide (such as a paraquat salt (for example paraquat dichloride or paraquat bis(methylsulphate), a diquat salt (for example diquat dibromide or diquat alginate) or glyphosate or a salt or ester thereof (such as glyphosate isopropylammonium, glyphosate sesquisodium or glyphosate trimesium (also known as sulfosate)), an insecticide or a fungicide. It is preferred that the water-soluble agrochemical is paraquat dichloride, diquat dibromide, glyphosate isopropylammonium or glyphosate trimesium (also known as sulfosate).

An electrolyte can be added to the composition to increase its ionic strength. The electrolyte helps to improve the insolubility of the material from which the sachet is made in the composition. (See Polyvinyl Alcohol - Properties and Applications pages 38-43, edited by C. A. Finch, published by J Wiley & Sons in 1973 and EP-A1-0518689.) Suitable electrolytes may, for example, comprise a cation or mixtures of cations selected from the list comprising: ammonium, copper, iron, magnesium, potassium and sodium; and an anion or mixture of anions selected from the list comprising: sulphate, nitrate, fluoride, chloride, bromide, iodide, acetate, tartrate, ammonium tartrate, benzenesulphonate, benzoate, bicarbonate, carbonate, bisulphate, bisulphite, sulphate, sulphite, borate, borotartrate, bromate, butyrate, chlorate, camphorate, chlorite, cinnamate, citrate, disilicate, dithionate, ethylsulphate, ferricyanide, ferrocyanide, fluorosilicate, formate, glycerophosphate, hydrogenphosphate, hydroxostannate, hypochlorite, hyponitrite, hypophosphite, iodate, isobutyrate, lactate, laurate, metaborate, metasilicate, methionate, methylsulphate, nitrite, oleate, orthophosphate, orthophosphite, orthosilicate, oxalate, perborate, perchlorate, phosphate, polyfluoride, polychloride, polyiodide, polybromide, polysulphide, polysulphate, polysulphite, salicylate, silicate, sorbate, stannate, stearate, succinate or valerate. Preferred electrolytes are ammonium sulphate, sodium sulphate, potassium sulphate, copper sulphate, ammonium nitrate, sodium nitrate, magnesium sulphate, potassium citrate, potassium nitrate, sodium chloride or potassium chloride.

Thus in another aspect the present invention provides an agrochemical composition packaged in a water-soluble or water-dispersible sachet, the agrochemical composition comprising a water-soluble, agrochemically active ingredient, an electrolyte, water and an agent to minimise water loss through the walls of the sachet.

The agrochemical composition may be in the form of a liquid (which may be thickened using known thickeners) or a gel (gelation may be achieved by using known methods, such as methods described in WO92/01377, EP-A1-0518689 or WO96/03038).

In another aspect the present invention provides an agrochemical composition packaged in a water-soluble or water-dispersible sachet, wherein the agent to minimise water loss through the walls of the sachet is a salt (especially an alkali metal salt) or ester (especially an alkyl ester) of an alkyl, alkenyl, phenyl or phenylalkyl acid (especially a carboxylic or sulphonic acid); an alcohol or an ether of an alcohol.

In a further aspect the present invention provides an agrochemical composition packaged in a water-soluble or water-dispersible sachet, wherein the agent to minimise water loss through the walls of the sachet is a salt (especially a sodium salt) or ester (especially C_{1-20} alkyl ester) of an alkyl, alkenyl or phenyl (wherein the phenyl group is optionally substituted with alkyl) acid (especially a carboxylic (such as oleic or adipic) or sulphonic (such as dodecylbenzene sulphonic) acid).

In a still further aspect the present invention provides an agrochemical composition packaged in a water-soluble or water-dispersible sachet, wherein the agent to minimise water loss through the walls of the sachet is a C_{1-12} alkyl ester of an alkyl or alkenyl carboxylic acid (for example methyl oleate, ethyl oleate or di-2-ethylhexyl adipate), or a C_{1-6} alkyl ester of an oil extracted from naturally occurring plants or seeds (such as methyl canolate (derived from canola oil) or methyl sunflowerate (derived from sunflower oil)).

Depending on the nature of the water-soluble, agrochemically active ingredient, one or more adjuvants or co-formulants (such as a wetter or anti-freezing agent) may also be comprised in the composition. Suitable adjuvants include neutral or anionic surfactants [such as a soap, a salt of an aliphatic monoester of sulphuric acid (for example, sodium lauryl sulphate), a salt of a sulphonated aromatic compound (for example, sodium dodecylbenzenesulphonate) or an alkyl glucoside] or polysaccharides. Suitable wetters include an alkyl glucoside (such as AL2042), a salt of a sulphonated aromatic compound (for example, sodium dodecylbenzenesulphonate), an alcohol ethoxylate or a diglucamide. Suitable thickeners include grades of xanthan gum (such as KELTROL® BT and KELZAN®).

In a further aspect the agrochemical composition also comprises a chelating or sequestering agent for calcium ions. A suitable chelating or sequestering agent is ethylenediaminetetraacetic acid (EDTA).

Over and above the components already mentioned, the agrochemical composition may also comprise an adhesive, an antifoaming agent, a buffer, a deodorant, a dye, an emetic, a preservative, an odourant, a perfume, a safener, a further solvent, a stabiliser, a synergist, a thickener or a wetting agent.

The water-soluble or water-dispersible sachet can be made from a variety of materials and preferred materials are polyethylene oxide, methyl cellulose or, especially, polyvinylalcohol (PVA). The PVA is generally partially or fully alcoholysed or hydrolysed, for example 40-100%, especially 80-100%, alcoholysed or hydrolysed polyvinyl acetate film. The PVA film may be a laminate of two or more thicknesses of film, a surface modified film (for example a film having a waxy layer) or a co-extruded film (such as is described in WO 94/29188). Preferred PVA films include M7030 (a monolayer film), L7030 (a laminate film), M7031 (a monolayer film), L7031 (a laminate film) and M9500 (also referred to as PXP2841, a monolayer film) all available from Chris Craft Industrial Products Inc. of South Holland, Illinois, USA.

The water-soluble or water-dispersible sachet can be formed and filled using standard techniques (such as thermoforming or vertical form-fill-sealing).

In use the packaged agrochemical composition is mixed with water (for example in an agrochemical sprayer) to give a sprayable solution of the water-soluble agrochemically active ingredient.

The packaged agrochemical composition of the present invention can be part of a bag-in-bag arrangement (for example as described in WO 92/17381 or WO 92/17383), part of an arrangement where two water-soluble sachets are joined at a common seal, part of an arrangement as described in Research Disclosure 38534 (published May 1996) or part of a bag-in-bag arrangement where the packaged agrochemical composition of the present invention is enclosed within a second water-soluble sachet holding only the packaged agrochemical composition of the present invention (the second sachet presenting a further barrier to contain the agrochemical composition).

Example 1 is an example of a composition that does not comprise an agent to minimise water loss through the walls of the sachet. Examples 2-8 illustrate the invention.

EXAMPLE 1

Component	Function	Concentration (g/l)
Paraquat dichloride	Active ingredient	200
Emetic	Emetic	0.5
AL2042 (Note 1)	Surfactant	100
"Dried" Magnesium sulphate	Electrolyte	140
KELTROL® BT (Note 2)	Gelling agent	3
SILCOLAPSE® 5020	Antifoam	1
SULFACIDE® blue 5J	Colour	5
Sodium hydroxide	pH adjuster	To pH 7
Water	Make-up solvent	To 1 litre

Note 1: AL2042 is a proprietary blend of alkyl polyglucoside and ethoxylated amine surfactants supplied by ICI Surfactants.

Note 2: KELTROL® BT is a salt tolerant grade of xanthan gum supplied by Kelco.

The composition was prepared by dissolving the paraquat dichloride and emetic in all but 25 ml of the total volume of water required. The KELTROL® BT was then added while stirring. Once fully dispersed the mixture was allowed to stand without stirring for 20 minutes to allow gel strength to build up. The AL2042, magnesium sulphate, SULFACIDE® blue and SILCOLAPSE® were then added sequentially with stirring. Sodium hydroxide (40% solution) was then added to adjust pH to 7. Finally, water was added, with mixing, to bring the volume up to 1l.

EXAMPLES 2-8

Component	Function	Concentration (g/l)
Paraquat dichloride	Active ingredient	200
Emetic	Emetic	0.5
AL2042	Surfactant	100
"Dried" Magnesium sulphate	Electrolyte	140
KELTROL® BT	Gelling agent	3
See TABLE I	Permeation inhibitor	50
SILCOLAPSE® 5020	Antifoam	1
SULFACIDE® blue 5J	Colour	5
Sodium hydroxide	pH adjuster	To pH 7
Water	Make-up solvent	To 1 litre

The composition was prepared by dissolving the paraquat dichloride and emetic in all but 75 ml of the total volume of water required. The KELTROL® BT was then added while stirring. Once fully dispersed the mixture was allowed to stand without stirring for 20 minutes to allow gel strength to build up. The AL2042, magnesium sulphate, SULFACIDE® blue and SILCOLAPSE® were then added sequentially with stirring. Sodium hydroxide (40% solution) was then added to adjust pH to 7. The permeation inhibitor was added with mixing. Finally, water was added, with mixing, to bring the volume up to 1l.

The premeation inhibitors are as in Table I.

TABLE I

EXAMPLE No.	PERMEATION INHIBITOR	AVAILABLE FROM
2	CRODAMOL® CAP	Croda Chemicals
3	Ethyl oleate	Croda Chemicals
4	NANSA® HS90S	Albright and Wilson
5	PRIOLUBE® 1403	Unichema International
6	Emery 2231	Henkel
7	Emery 2230	Henkel
8	CRODAMOL® DOA	Croda Chemicals

CRODAMOL® CAP is a proprietary blend of branched chain esters

NANSA® HS90S is sodium dodecylbenzene sulphonate (90% active)

Emery 2231 is a methylated canola oil

Emery 2230 is a methylated sunflower oil

PRIOLUBE® 1403 is methyl oleate

CRODAMOL® DOA is di-2-ethylhexyl adipate

EXAMPLE 9

A quantity (around 50g) of each of the compositions from Examples 1-8 was packed into a water-soluble sachet made from M7030 grade polyvinyl alcohol film (76 micron thickness, supplied by Chris Craft). Each packed water-soluble sachet was uniquely labelled and accurately weighed on an analytical balance. Each packed, weighed sachet was placed in an individual polythene/aluminium/paper secondary pack which was sealed. Two sealed packs of each composition were placed in a constant temperature oven at 40°C and stored for 4 weeks.

Following storage each pack was allowed to reach ambient laboratory conditions. The secondary pack was carefully opened and any signs of permeation of the water-soluble sachet noted. The external surface of the sachet was wiped, the water-soluble sachet was re-weighed, and the % weight loss calculated. The findings are presented in TABLE II.

TABLE II

EXAMPLE No.	Replicate No.	Initial Weight (g)	Final Weight (g)	Weight Loss (%)
1 *	1	51.6636	51.2150	0.87
1 *	2	52.8301	52.1413	1.30
2 *	1	49.3533	49.2215	0.27
2 *	2	48.2992	48.1415	0.32
3 *	1	46.6707	46.4283	0.52
3 *	2	45.7361	45.5910	0.32
4 *	1	49.2898	49.0820	0.42
4 *	2	51.0974	50.8878	0.41
5 *	1	49.5055	49.3089	0.40
5 *	2	50.3473	50.1688	0.35
5 **	1	51.8850	51.7806	0.20
5 **	2	52.5036	52.3752	0.24
6 *	1	47.5367	47.3411	0.41
6 *	2	52.0131	51.7638	0.48
7 *	1	47.9182	47.7441	0.36
7 *	2	56.3024	56.1232	0.32
8 *	1	53.9247	53.7520	0.32
8 *	2	49.1725	49.0110	0.33

* Shiny side of film (forming water-soluble sachet) is on the inner surface of the sachet.

** Shiny side of film (forming water-soluble sachet) is on the outer surface of the sachet.

EXAMPLE 10

A composition containing the following components was prepared using the method described for Examples 2-8.

Component	Function	Concentration (g/l)
Paraquat dichloride	Active ingredient	200
Emetic	Emetic	0.5
AL2042	Surfactant	100
Magnesium sulphate hydrate ($MgSO_4.7H_2O$)	Electrolyte	234
KELTROL® BT	Gelling agent	3
PRIOLUBE® 1403	Permeation inhibitor	50
SILCOLAPSE® 5020	Antifoam	1
SULFACIDE® blue 5J	Colour	5
Sodium hydroxide	pH adjuster	To pH 6.5-7
Water	Make-up solvent	To 1 litre

Quantities (around 50g) of this composition were packaged in water-soluble sachets of various films and stored according to the methodology described in Example 9. The L7030 film was 76 microns thick while the M7031 and M9500 films were both 50 microns thick. The results are shown in TABLE III.

TABLE III

Film	Replicate no.	Initial weight (g)	Final weight (g)	Weight Loss (%)
L7030	1	51.2962	51.1689	0.25
L7030	2	53.2853	53.1292	0.29
M7031	1	49.9788	49.8286	0.30
M7031	2	50.5838	50.4297	0.30
M9500	1	50.1853	49.9749	0.42
M9500	2	49.6982	49.4956	0.41

EXAMPLE 11

A composition containing the following components was prepared using the method described for Examples 2-8.

Component	Concentration (g/l)
Diquat dibromide	140
AL2042	100
Magnesium sulphate hydrate ($MgSO_4.7H_2O$)	234
KELTROL® BT	3
SILCOLAPSE® 5020	1
SULFACIDE® blue 5J	5
Methyl oleate (permeation inhibitor)	50
Sodium hydroxide (40% solution)	to pH 6.5-7.0
Water	to 1 litre

Quantities (around 50g) of this composition were packaged in three water-soluble sachets of M7031 film (50 microns thick) and stored according to the methodology described in Example 9. At the end of four weeks it was found that the weight loss (%) for each of the three sachets was: 0.16, 0.04 and 0.08.

When methyl oleate was omitted from the composition, and that composition was packed and stored as described above, it was found that the weight loss (%) for each of the three sachets was: 2.67, 2.45 and 1.7.

EXAMPLES 12 and 13

Table IV presents the components for two compositions containing the components listed in the amounts shown. The compositions were prepared using the method described for Examples 2-8. Quantities (around 50g) of these compositions were packaged (in triplicate) in water-soluble sachets of M7031 film (50 microns thick) and stored according to the methodology described in Example 9. At the end of four weeks the weight loss (%) for each sachet was assessed. The bracketed weight loss data represent weight losses (%) for the situation where methyl oleate was omitted from the compositions.

TABLE IV

Example No.	12	13
Component		
Sulfosate (g)	300	
Glyphosate (g)		240
Al2042 (g)	100	
Ammonium sulphate (g)	241	241
KELTROL® BT (g)	3	3
SILCOLAPSE® 5020 (g)	1	1
SULFACIDE® blue 5J (g)	5	5
Methyl oleate (g)	50	50
Water	to 1 litre	to 1 litre
Weight Loss (%)		
Replicate a	0.02 (0.08)	0.02 (0.16)
Replicate b	0.00 (0.06)	0.10 (1.23)
Replicate c	0.04 (0.12)	0.08 (0.31)

EXAMPLES 14-20

Table V presents the components for compositions containing the components listed in the amounts shown. The compositions were prepared using the method described for Examples 2-8. Quantities (around 50g) of these compositions were packaged (in triplicate) in water-soluble sachets of M7031 film (50 microns thick) and stored according to the methodology described in Example 9. At the end of four weeks the weight loss (%) for each sachet was assessed. Bracketed weight loss data represent weight losses (%) for the situation where the permeation inhibitor was omitted from certain compositions.

TABLE V

Example No.	14	15	16	17	18	19	20
Component							
Paraquat dichloride (g)	200	200	200	200	200	200	200
AL2042 (g)	100	100	100	100	100	100	100
Magnesium sulphate $7H_2O$ (g)	234		234	234	234	234	
Ammonium sulphate (g)		234					
Tripotassium citrate (g)							350
KELTROL® BT (g)			3	3		3	
KELZAN® (g)	3	3					3
SILCOLAPSE® 5020 (g)	1	1	1	1	1	1	1
SULFACIDE® blue 5J (g)	5	5	5	5	5	5	5
Methyl oleate (g) (permeation inhibitor)	50	50					
ISOPAR® M (g) (permeation inhibitor)			50		50		50
Heated methyl oleate * (g) (permeation inhibitor)				50			
Methyl oleate + EDTA (g) (permeation inhibitor + chelating agent)						50	
Sodium hydroxide 40% solution	to pH6.5-7.0	to pH6.5-7.0	to pH6.5-7.0	to pH6.5-7.0	to pH6.5-7.0	to pH6.5-7.0	to pH6.5-7.0
Water	to 1 litre	to 1 litre	to 1 litre	to 1 litre	to 1 litre	to 1 litre	to 1 litre

* Heated at 100°C for 7 days

TABLE V (Continued)

Example No.	14	15	16	17	18	19	20
Weight Loss (%)							
Replicate a	0.02	0.11 (0.42)	0.02	0.04	0.09	0.2	0.1 (0.21)
Replicate b	0.04	0.06 (0.53)	0.06	0.04	0.08	0.07	0.02 (0.18)
Replicate c	0	0.15 (0.49)	0.06	0.06	0.08	0.08	0.02 (0.19)

CLAIMS

1. An agrochemical composition packaged in a water-soluble or water dispersible sachet, the agrochemical composition comprising a water-soluble, agrochemically active ingredient, water and an agent to minimise water loss through the walls of the sachet.

2. An agrochemical composition packaged in a water-soluble sachet as claimed in claim 1, the agrochemical composition comprising a water-soluble, agrochemically active ingredient, an electrolyte, water and an agent to minimise water loss through the walls of the sachet.

3. An agrochemical composition packaged in a water-soluble or water-dispersible sachet as claimed in claim 1 or 2, wherein the agent to minimise water loss through the walls of the sachet is an alkyl, alkenyl, aryl or arylalkyl acid or a salt or ester thereof; an ester of a naturally occurring oil; a mineral or synthetic oil; an alcohol, an ether of an alcohol or a glyceride.

4. An agrochemical composition packaged in a water-soluble or water-dispersible sachet as claimed in claim 1, 2 or 3, wherein the agent to minimise water loss through the walls of the sachet is an ester of an alkyl, alkenyl, aryl or arylalkyl acid; an ester of a naturally occurring oil; or a mineral or synthetic oil.

5. An agrochemical composition packaged in a water-soluble or water-dispersible sachet as claimed in claim 1, 2 or 3, wherein the agent to minimise water loss through the walls of the sachet is a salt or ester of an alkyl, alkenyl or phenyl (wherein the phenyl group is optionally substituted with alkyl) acid.

6. An agrochemical composition packaged in a water-soluble or water-dispersible sachet as claimed in any one of the preceeding claims, wherein the agent to minimise water loss through the walls of the sachet is a C_{1-12} alkyl ester of an alkyl or alkenyl carboxylic acid or a C_{1-6} alkyl ester of an oil extracted from naturally occurring plants or seeds.

7. An agrochemical composition packaged in a water-soluble or water-dispersible sachet as claimed in any one of the preceeding claims, wherein the agent to minimise water loss through the walls of the sachet is methyl oleate, ethyl oleate, di-2-ethylhexyl adipate, methyl canolate or methyl sunflowerate.

8. An agrochemical composition packaged in a water-soluble or water-dispersible sachet as claimed in any one of the preceeding claims, wherein the sachet is made of partially or fully alcoholysed or hydrolysed polyvinyl acetate film.

9. An agrochemical composition packaged in a water-soluble or water-dispersible sachet obtained by packing a composition comprising a water-soluble, agrochemically active ingredient, water and an agent to minimise water loss through the walls of the sachet in a water-soluble or water-dispersible sachet.

INTERNATIONAL SEARCH REPORT

Inte. onal Application No
PCT/GB 97/00001

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A01N25/34 A01N25/04 A01N57/20 A01N43/90 A01N43/40
B65D85/82 //(A01N57/20,25:34,25:04),(A01N43/90,25:34,25:04),
(A01N43/40,25:34,25:04)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A01N B65D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	WO 96 03038 A (ZENECA LTD ;TADROS THARWAT FOUAD (GB); TAYLOR PHILIP (GB)) 8 February 1996 see page 4, line 19-31; examples 1-19	1-5,8,9
X	EP 0 518 689 A (RHONE POULENC AGROCHIMIE) 16 December 1992 cited in the application see the whole document	1-9
Y	EP 0 199 034 A (SCHERER GMBH R P ;THEURER KARL EUGEN (DE)) 29 October 1986 see page 3, line 4 - page 4, line 5; example 2	1-9
Y	US 3 892 905 A (ALBERT ROBERT EYER) 1 July 1975 see column 4, line 1 - line 44	1-9

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&" document member of the same patent family

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Date of the actual completion of the international search	Date of mailing of the international search report
18 April 1997	22.05.97

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax (+ 31-70) 340-3016	Klaver, J

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INTERNATIONAL SEARCH REPORT

information on patent family members

Inte ɔnal Application No
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